

=> d his

(FILE 'HOME' ENTERED AT 14:33:24 ON 22 NOV 2004)

FILE 'REGISTRY' ENTERED AT 14:33:32 ON 22 NOV 2004

L1 STRUCTURE UPLOADED

L2 STRUCTURE UPLOADED

L3 0 S L1 OR L2

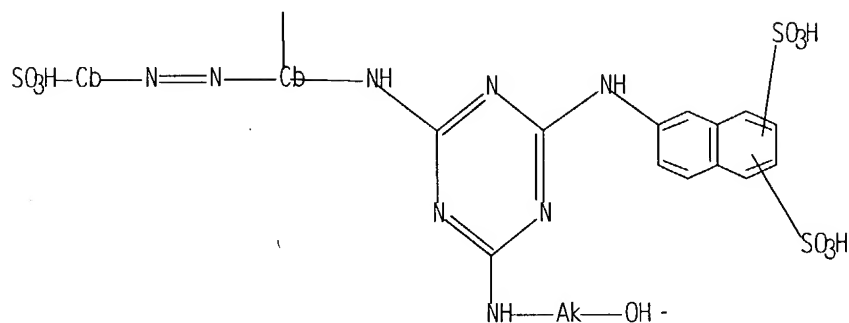
L4 5 S L3 FULL

FILE 'CAPLUS' ENTERED AT 14:34:38 ON 22 NOV 2004

L5 2 S L4

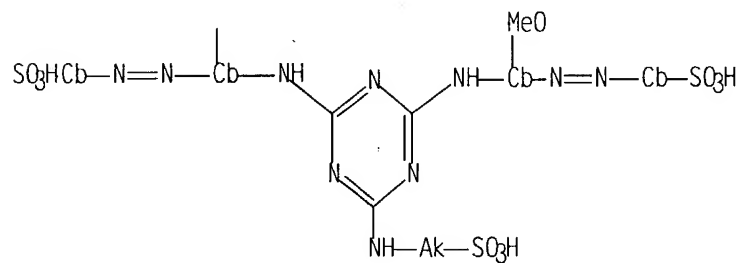
=> d que 15 stat

L1 STR



Structure attributes must be viewed using STN Express query preparation.

L2 STR



Structure attributes must be viewed using STN Express query preparation.

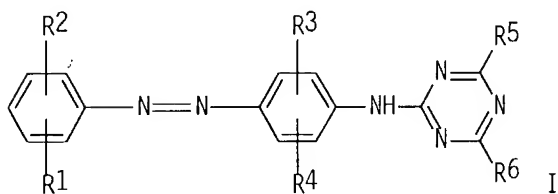
L4 5 SEA FILE=REGISTRY SSS FUL L1 OR L2

L5 2 SEA FILE=CAPLUS ABB=ON PLU=ON L4

=> d 1-2 bib abs hitstr

L5 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2004 ACS on STN
 AN 2004:293437 CAPLUS
 DN 140:322866
 TI Yellow dyes and ink compositions
 IN Holloway, Ann P.; Feeman, James Frederic
 PA USA
 SO U.S. Pat. Appl. Publ., 18 pp.
 CODEN: USXXCO
 DT Patent
 LA English
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2004068102	A1	20040408	US 2002-265547	20021007
PRAI	US 2002-265547		20021007		
OS	MARPAT 140:322866				
GI					



AB Provided are water-soluble yellow azo dyes (I; R1 = carboxy, sulfo, or Li or Na or K salts or amino derivs.; R2 = H, halogen, organic group; R3 = H, organic group; R4 = H, alkyl, alkoxy; aromatic amino group; R5, R6 = organic amino group) for jet ink compns. The dyes and ink compns. of the present invention exhibit a good balance of optical d., print reliability, water fastness, and light fastness and show improved water solubility, thus rendering the ink compns. to be especially suitable for print heads having small nozzle openings. Examples with 3 dyes were given.

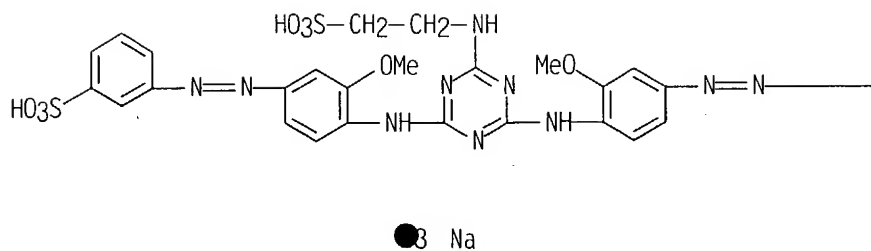
IT 677301-02-9 677301-03-0

RL: TEM (Technical or engineered material use); USES (Uses)
 (yellow azo dyes and jet-printing inks ink compns. containing them)

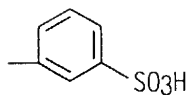
RN 677301-02-9 CAPLUS

CN Benzenesulfonic acid, 3,3'-[[6-[(2-sulfoethyl)amino]-1,3,5-triazine-2,4-diyl]bis[imino(3-methoxy-4,1-phenylene)azo]]bis-, trisodium salt (9CI)
 (CA INDEX NAME)

PAGE 1-A

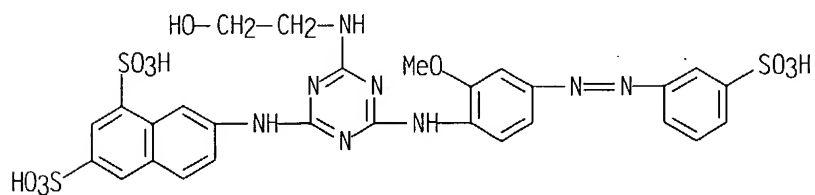


PAGE 1-B



RN 677301-03-0 CAPLUS

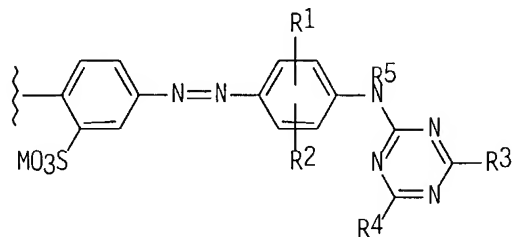
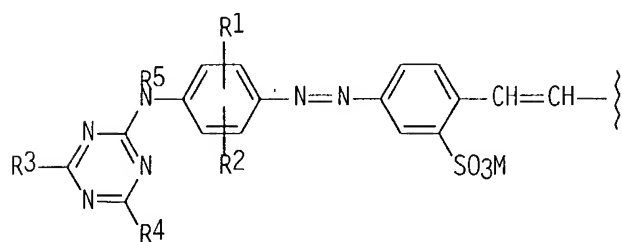
CN 1,3-Naphthalenedisulfonic acid, 7-[[4-[(2-hydroxyethyl)amino]-6-[[2-methoxy-4-[(3-sulfo)phenyl]azo]phenyl]amino]-1,3,5-triazin-2-yl]amino]-, trisodium salt (9CI) (CA INDEX NAME)



● 3 Na

L5 ANSWER 2 OF 2 CAPLUS COPYRIGHT 2004 ACS on STN
 AN 1995:716835 CAPLUS
 DN 123:85950
 TI Disazo dyes of the stilbene series, their preparation and use
 IN Pedrazzi, Reinhard; Zirngibl, Ulrich
 PA Sandoz Ltd., Switz.; Sandoz-Patent-GmbH
 SO Eur. Pat. Appl.. 51 pp.
 CODEN: EPXXDW
 DT Patent
 LA English
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 651027	A1	19950503	EP 1994-810617	19941026
	EP 651027	B1	19990818		
	R: BE, CH, DE, ES, FR, GB, IT, LI, NL, SE				
	DE 4337181	A1	19950504	DE 1993-4337181	19931030
	US 5495003	A	19960227	US 1994-330292	19940227
	ES 2137344	T3	19991216	ES 1994-810617	19941026
	CA 2134487	AA	19950501	CA 1994-2134487	19941027
	BR 9404282	A	19950704	BR 1994-4282	19941027
	JP 07196936	A2	19950801	JP 1994-263635	19941027
PRAI	DE 1993-4337181	A	19931030		
OS	MARPAT 123:85950				
GI					



I

AB Disazo dyes of the general formula I (each M = H, cationic group; each R1 = H, halogen, amino, OH, alkyl, alkoxy, aryl, aryloxy; each R2 = H, halogen, CN, CO2H, SO3H, organic group; each R3 = F, Cl, SO3H, alkylsulfonyl, arylsulfonyl; each R4 = substituted amino; each R5 = H, organic group; R1 may combine with R2 or R5 to complete a ring) are suitable for dyeing or printing organic substrates containing OH, SH, or amide groups, especially cotton or

paper. Thus, 4,4'-diaminostilbene-2,2'-disulfonic acid was tetrazotized and coupled with 2 mol $\text{PhNHCH}_2\text{SO}_3\text{H}$, and the product was deprotected with 30% NaOH and condensed with 2,4-dichloro-6-(6,8-disulfo-2-naphthylamino)-s-triazine to give a I. λ_{max} 426 nm in 50% aqueous DMF, which gave lightfast golden yellow shades on paper and cotton.

IT 165738-66-9P

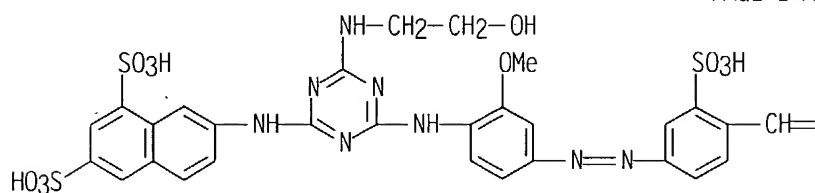
RL: IMF (Industrial manufacture); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses)

(yellow; preparation of stilbene derivative disazo dyes for cotton and paper)

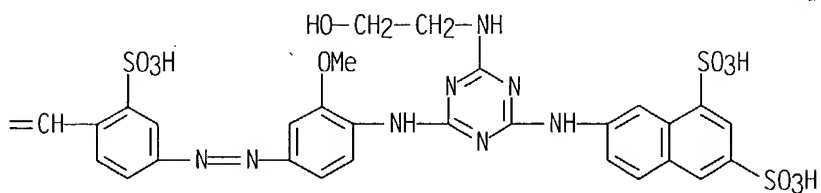
RN 165738-66-9 CAPLUS

CN 1,3-Naphthalenedisulfonic acid, 7,7'-[1,2-ethenediylbis[(3-sulfo-4,1-phenylene)azo(2-methoxy-4,1-phenylene)imino[6-[(2-hydroxyethyl)amino]-1,3,5-triazine-4,2-diyl]imino]]bis- (9CI) (CA INDEX NAME)

PAGE 1-A



PAGE 1-B



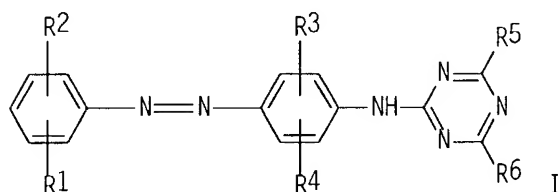
=> => d que 19 stat

L6 7 SEA FILE=CAPLUS ABB=ON PLU=ON "HOLLOWAY ANN P"/AU
L7 54 SEA FILE=CAPLUS ABB=ON PLU=ON ("FEEMAN JAMES F"/AU OR
"FEEMAN JAMES FREDERIC"/AU)
L8 56 SEA FILE=CAPLUS ABB=ON PLU=ON L6 OR L7
L9 13 SEA FILE=CAPLUS ABB=ON PLU=ON L8 AND YELLOW

=> d 1-13 bib abs

L9 ANSWER 1 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN
 AN 2004:293437 CAPLUS
 DN 140:322866
 TI Yellow dyes and ink compositions
 IN Holloway, Ann P.; Feeman, James Frederic
 PA USA
 SO U.S. Pat. Appl. Publ., 18 pp.
 CODEN: USXXCO
 DT Patent
 LA English
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2004068102	A1	20040408	US 2002-265547	20021007
PRAI	US 2002-265547		20021007		
OS	MARPAT 140:322866				
GI					



AB Provided are water-soluble **yellow** azo dyes (I; R1 = carboxy, sulfo, or Li or Na or K salts or amino derivs.; R2 = H, halogen, organic group; R3 = H, organic group; R4 = H, alkyl, alkoxy; aromatic amino group; R5, R6 = organic amino group) for jet ink compns. The dyes and ink compns. of the present invention exhibit a good balance of optical d., print reliability, water fastness, and light fastness and show improved water solubility, thus rendering the ink compns. to be especially suitable for print heads having small nozzle openings. Examples with 3 dyes were given.

L9 ANSWER 2 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN
AN 1998:619019 CAPLUS
DN 129:218065
TI Ink jet ink dyes based on ammonium containing cationic dyes having
improved color migration and fading properties and dye manufacture
IN Feeman, James Frederic; Sun, Jing Xiao
PA Lexmark International Inc. USA
SO Brit. UK Pat. Appl., 48 pp.
CODEN: BAXXDU
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	-----	---	-----	-----	-----
PI	GB 2315493	A1	19980204	GB 1997-14797	19970714
	GB 2315493	B2	20010103		
	GB 2346618	A1	20000816	GB 2000-10797	19970714
	GB 2346618	B2	20010207		
	US 5969112	A	19991019	US 1998-55007	19980403
	US 5998590	A	19991207	US 1998-54606	19980403
	US 6130319	A	20001010	US 1998-55008	19980403
PRAI	US 1996-690467	A	19960724		
	GB 1997-14797	A3	19970714		

OS MARPAT 129:218065

AB Phthalocyanine and azo dye derivs. modified with cationic groups are useful in jet inks. Thus, 0.01125 mol C.I. Reactive Red 180 was heated with 0.01125 mol diethanolamine at 50° at pH 7.5 for 18 h and treated with di-Me sulfate to give the cationic dye, which was added (2%), with 2,2-thiodiethanol 15, 1,2-hexanediol 6, Proxel GXL 0.1%, and the balance H2O to give a jet ink for printing images with good lightfastness (72 h, Xenon Arc Fadometer).

L9 ANSWER 3 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN
AN 1998:220856 CAPLUS
DN 128:271816
TI Ink system with reduced bleed
IN **Feeman, James F.; Holloway, Ann P.**; Zimmer, Agnes K.;
Sun, Jing X.; Franey, Terence E.; Mrvos, James M.; Beach, Bradley L.
PA Lexmark International, Inc., USA
SO U.S., 24 pp.
CODEN: USXXAM
DT Patent
LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 5735941	A	19980407	US 1996-690468	19960724
	GB 2315759	A1	19980211	GB 1997-15666	19970724
	GB 2315759	B2	20000705		
PRAI	US 1996-690468	A	19960724		

OS MARPAT 128:271816

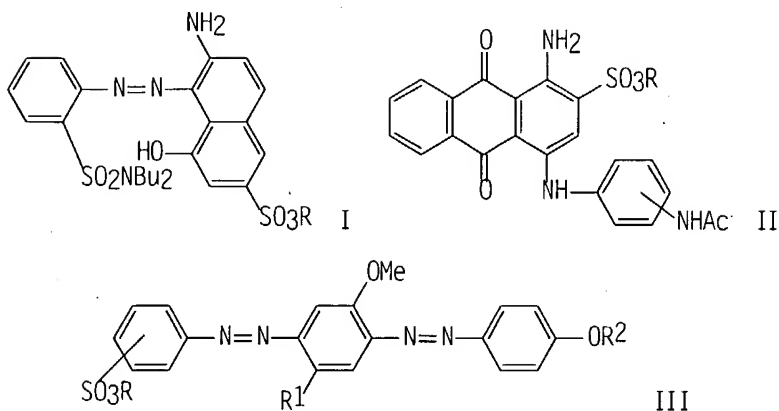
AB An ink system comprising a first ink containing a flocculating dye in an aqueous solution and a second ink containing a dispersant-pigment complex in an aqueous solution reduces bleed between the two inks when they are applied side by side, wherein the flocculating dye flocculates the dispersant-pigment complex. A first ink comprised Basic **Yellow 45**, 2,2'-thiodiethanol, 1,2-hexanediol, Proxel GXL, and deionized water, and a second ink comprised carbon black, methacrylic acid-monomethacryloyloxypropyl-terminated di-Me siloxane-stearyl methacrylate terpolymer, and deionized water.

RE.CNT 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 4 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN
 AN 1986:35418 CAPLUS
 DN 104:35418
 TI Dyeing polyamide fibers
 IN Rowe, Jay E.; Feeman, James F.
 PA Crompton and Knowles Corp., USA
 SO Eur. Pat. Appl., 18 pp.
 CODEN: EPXXDW
 DT Patent
 LA English
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 153052	A1	19850828	EP 1985-300638	19850130
	EP 153052	B1	19880406		
	R: BE, CH, DE, FR, GB, IT, LI, NL				
	US 4579561	A	19860401	US 1985-690010	19850109
PRAI	US 1984-575573	A	19840131		
	US 1985-690010	A	19850109		

GI



AB Polyamide fibers, especially carpeting, are dyed with a trichromatic dye system comprising red acid dyes [I, R = Na, K, Li, or N(R₃)₄ where R₃ is H or [CH₂CH(OR₄)_n]H and R₄ is H, Me, or Et, and n = 1, 2, 3, or 4], blue acid dyes [II, R as previously defined], and **yellow** acid dyes [III, R as previously defined, R₁ = H, Me; R₂ = Na, Me, CH₂CH₂OH, CH₂CHMeOH]. A dye mixture was prepared from (I, R = Na) [99740-46-2] 2.2, and (II, R = Na) [70571-81-2] 1.0, and (III, R = Na, R₁ = R₂ = Me) [68555-86-2] 2.5 g and continuously dyed a 40-oz sample of 100% nylon 66 carpeting at a running speed equivalent to 5 yd/min. A level-dyed carpet of medium brown shade having excellent fastness to light and atmospheric gases was obtained.

L9 ANSWER 5 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN
AN 1976:107065 CAPLUS
DN 84:107065
TI Disazo disulfonic acids
IN Feeman, James F.
PA Crompton and Knowles Corp., USA
SO U.S., 5 pp.
CODEN: USXXAM
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 3932376	A	19760113	US 1971-187150	19711006
PRAI	US 1971-187150		19711006		

GI For diagram(s), see printed CA Issue.

AB Six disazo dyes (I, R, R₂ = H, Me; R₁ = H, MeO; R₃ = H, Me, Et, SO₂C₆H₄Cl-4; n = 1, 2; m + n = 2) were prepared and dyed polyamide fibers fast reddish **yellow** to orange shades from a weakly acid dyebath while reserving cationic-dyeable polyamide fibers. Thus, 4-HO₃SC₆H₄N:NC₆H₃(SO₃H)NH₂-3,4 [101-50-8] was diazotized and coupled with PhOH [108-95-2] to give I (R = R₁ = R₂ = R₃ = H; n = 1 (p-substituted); m = 1) [58480-36-7]. The other I were similarly prepared.

L9 ANSWER 6 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN
 AN 1974:427188 CAPLUS
 DN 81:27188
 TI Disazo dyes
 IN **Feeman, James F.**
 PA Crompton and Knowles Corp.
 SO Ger. Offen., 15 pp.
 CODEN: GWXXBX
 DT Patent
 LA German
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	-----	----	-----	-----	-----
PI	DE 2336869	A1	19740214	DE 1973-2336869	19730719
	CA 999854	A1	19761116	CA 1973-169803	19730430
	ES 415501	A1	19760216	ES 1973-415501	19730602
	BE 800425	A1	19731204	BE 1973-131853	19730604
	ZA 7304325	A	19740529	ZA 1973-4325	19730626
	JP 49059130	A2	19740608	JP 1973-74467	19730703
	CH 587312	A	19770429	CH 1973-10048	19730710
	GB 1390706	A	19750416	GB 1973-33685	19730716
	NL 7310179	A	19740123	NL 1973-10179	19730720
	FR 2193814	A1	19740222	FR 1973-26811	19730720
	IT 998261	A	19760120	IT 1973-26845	19730720

PRAI US 1972-273767 19720721

AB Azo compound I(R = R1 = H) and azo compound I(R = MeO, R1 = Me)(II), fast **yellow** and orange dyes resp. for deep-dyeable nylon fibers, were prepared Thus, successive reaction of diazotized 2-amino-4,8-naphthalenedisulfonic acid with cresidine, diazotization, reaction with phenol, and alkylation with Et2SO4 gave II. The other dye was similarly prepared

L9 ANSWER 7 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN
 AN 1973:85902 CAPLUS
 DN 78:85902
 TI Alkali-fast disazo dyes
 IN Feeman, James F.
 PA Crompton and Knowles Corp.
 SO Ger. Offen., 21 pp.
 CODEN: GWXXBX
 DT Patent
 LA German
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 2215789	A	19721207	DE 1972-2215789	19720330
	DE 2215789	C2	19820401		
	US 4166059	A	19790828	US 1971-130850	19710402
	NL 7204382	A	19721004	NL 1972-4382	19720330
	NL 173971	B	19831101		
	NL 173971	C	19840402		
	FR 2132255	A5	19721117	FR 1972-11284	19720330
	FR 2132255	B1	19760806		
	CA 975357	A1	19750930	CA 1972-138685	19720330
	CH 571558	A	19760115	CH 1972-4806	19720330
	BE 781525	A1	19720717	BE 1972-115808	19720331
	IT 954828	A	19730915	IT 1972-22658	19720331
	JP 57015143	B4	19820329	JP 1972-31790	19720331
	ES 401400	A1	19750901	ES 1972-401400	19720401
	GB 1373141	A	19741106	GB 1972-15486	19720404
PRAI	US 1971-130850		19710402		
	US 1971-130598		19710402		

AB Three water-soluble disazo dyes [I, R = H, OMe, R1 = H, Me, OMe R2 = H, Me] were prepd and were used to dye nylon, silk, and wool **yellow** to red shades of good light-, wash-, and alkalifastness. Thus, 2,5-(MeO)2C6H3NH2 was coupled with diazotized 4-MeC6H4SO2NHSO2C6H4NH2-3 to give the azo intermediate, which was diazotized, coupled with PhOH, and etherified with Et2SO4 to give disazo dye I(R = R1 = MeO, R2 = H, 3-substituted.) [38775-62-1].

L9 ANSWER 8 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN
AN 1971:88658 CAPLUS
DN 74:88658
TI 2-(5-Hydroxy-3-methyl-1-phenyl-3-pyrazolylazo)naphthalene-1-sulfonate dyes
IN **Feeman, James F.**
PA Crompton and Knowles Corp.
SO Ger. Offen., 9 pp.
CODEN: GWXXBX
DT Patent
LA German
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	-----	----	-----	-----	-----
PI	DE 2010712	A	19701223	DE 1970-2010712	19700306
	GB 1239548	A	19710714	GB 1970-1239548	19700202
	BE 745761	A	19700810	BE 1970-745761	19700210
	FR 2034686	A5	19701211	FR 1970-4992	19700212
	FR 2034686	B1	19730316		
	CH 523952	A	19720615	CH 1970-523952	19700227
PRAI	US 1969-804997		19690306		
GI	For diagram(s), see printed CA Issue.				
AB	The title compds. I (R = Cl or Me), prepared by coupling diazotized 2,1-H ₂ NC10H ₆ SO ₃ H with the corresponding 1-phenyl-3-methyl-2-pyrazolin-5-one, gave bright yellow shades of good fastness on polyamides.				

L9 ANSWER 9 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN
 AN 1968:437098 CAPLUS
 DN 69:37098
 TI 3-(2-Chloroethylsulfonyl) benzenesulfonyl chlorides
 IN Feeman, James F.
 PA Crompton and Knowles Corp.
 SO Fr., 6 pp.
 CODEN: FRXXAK
 DT Patent
 LA French
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	FR 1487789		19670707		
	GB 1151545			GB	
PRAI	US		19650816		

GI For diagram(s), see printed CA Issue.

AB The title compds. (I), useful for manufacturing fiber-reactive dyes, are prepared by chlorosulfonation of 2-chloroethyl phenyl sulfones at high temperature. Thus, 204.5 parts $\text{PhSO}_2\text{CH}_2\text{CH}_2\text{Cl}$ in 250 parts ClSO_3H was heated to 135° in 1 hr., kept at 135° for 3 hrs., and cooled to 35° . SOCl_2 (120 parts) was added, the mixture brought to 135° in 1 hr., kept at 135° for 3 hrs., cooled to 25° , and poured into a H_2O -ice mixture. The crystalline mass was ground, washed (cold H_2O) neutral, and dried to give 270 parts I (R = H) (II), m. $68-9^\circ$ (AcOH). Similarly other I were prepared (R, m.p. of starting sulfone, and m.p. of I given): 4-Me, -(oil), $135-6^\circ$; 5-Me, -(oil), $123-5^\circ$; 6-Me, $77-9^\circ$, $117-19^\circ$; 4-Et, -(oil), $117-18^\circ$; 4-iso-Pr, -(oil), $117-19^\circ$; 4-Cl, $57-8^\circ$, $129-31^\circ$; 5-Cl, $60-1^\circ$, -(oil); 6-Cl, $98-9^\circ$, $116-19^\circ$. AcONa (10 parts) and 31 parts II were added to a neutral solution of 18.8 parts 2,4-(H₂N)₂C₆H₃SO₃H in 200 parts H_2O at 25° , the mixture stirred for 18 hrs. at $25-30^\circ$, heated at 70° until complete dissoln., clarified, cooled and salted with 20 parts NaCl to give 3,4-H₂N(HO₃S)₂C₆H₃NHSO₂C₆H₄SO₂CH₂CH₂Cl-3 (III). III, diazotized and coupled with 1-phenyl-3-methyl-5-pyrazolone, gave a yellow dye for nylon. Similarly, other fiber-reactive dyes were prepared from III (coupling component and shade on cotton given): 1-(p-sulfophenyl)-3-methyl-5-pyrazolone, yellow; 1,3,6-HOC₁₀H₅(SO₃H)₂, orange-red; 2,3,6-H₂NC₁₀H₅(SO₃H)₂, orange; 1,3,7-HO(HO₃S)₂C₁₀H₅NH₂ (IV), red; 1,3-HOC₁₀H₆SO₃H, orange; 2,3,6-HOC₁₀H₅(SO₃H)₂, red. The 2:1 Cr complex of V (43.2 parts) was dissolved at pH 6.5 in 300 parts H_2O and 5 parts NaHCO_3 , 15 parts II added in 30 min. at $70-5^\circ$, the mixture kept at 75° and salted with 8 parts NaCl to give a bluish-gray dye for nylon. Similarly, other fiber-reactive dyes were prepared from II (starting dye and shade given): VI (Pc = phthalocyanine), bright turquoise-blue on cotton; 1-amino-4-(p-hydroxyanilino)-2-anthraquinonesulfonic acid, bright blue on nylon; 0-HO₃SC₆H₄NH₂ → IV, red on cellulose; Cu complex of 2,5-HO(HO₃S)₂C₆H₃NH₂ → 1,3,6-HO(HO₃S)₂C₁₀H₅NH₂, bluish red on cotton; 4-HOC₆H₄NH₂ → 1-(2-chloro-5-sulfophenyl)-3-methyl-5-pyrazolone, bright yellow on nylon; 2,4-O₂N(HO₃S)₂C₆H₃NHC₆H₄OH-4, bright yellow on nylon; 3-HO₃SC₆H₄NH₂ → 1-C₁₀H₇NH₂ → PhOH, orange on nylon.

L9 ANSWER 10 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN
 AN 1967:38845 CAPLUS
 DN 66:38845
 TI Sulfomethylated bisphenol-formaldehyde compositions for treating wool
 IN Feeman, James F.
 PA Crompton and Knowles Corp.
 SO U.S., 4 pp.
 CODEN: USXXAM
 DT Patent
 LA English
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 3293214		19661220	US	19620515

GI For diagram(s), see printed CA Issue.
 AB Bisphenol-HCHO condensates of the general formula I, where R is Et or Me, R1R1 is a pentamethylene group, R2 is H or Me, m is 1-6, and X represents the substitution of 1-2 sulfomethyl groups per 4 benzene nuclei in the chain, were prepared and used to reduce wool felting and shrinking. In the structure, each R2 represents H or Me (the same or different). Bisphenol was condensed with HCHO in alkali, acidified, heated to reflux, redissolved in aqueous alkali, and treated with a I-bisulfite adduct (II). Thus, bisphenol A 456 and NaOH 80 were dissolved in H2O 1000 parts and 81 parts 37% HCHO solution was added. After 2 hrs. at 90° and subsequent addition of 265 parts HCl, the mixture separated into 2 phases. Heating 3 hrs. at 95° and pH 1-1.5 and addition of 160 parts of 50% NaOH redissolved the viscous layer. II (prepared from 37% HCHO 162, H2O 165, and Na2S2O5 200 parts) was added and the solution heated at 95° for 18 hrs. After cooling, the pH was adjusted to 6 with 420 parts HCl and 200 parts NaCl was added. Filtration and drying gave 710 parts of a H2O-soluble colorless solid (III). A laundering bath was prepared by dissolving III 4 and Na dodecylbenzenesulfonate (IV) 2 in H2O 2000 parts at 120°F. Soiled wool garments were washed for 15 min., rinsed, spun dry, and dried. The wool garment was free of soil and felt shrinkage, while a control treated with IV alone was soiled and showed signs of felting. After 6 cycles, the control was felted and unusable, while the treated sample was in good condition. Dyeing of 380 parts wool fabric with C.I. Acid Yellow 40 2.28, C.I. Acid Red 114 0.76, C.I. Acid Blue 78 0.76, I 15.2, HOAc 7.6, and H2O 9500 parts at 180° gave a uniform brown shade with no felting, while a control was badly felted. Similar compns. were prepared from 2,2-bis(4-hydroxy-3-methylphenyl)propane, 1,1-bis(4-hydroxyphenyl)cyclohexane, and 2,2-bis(p-hydroxyphenyl)butane.

L9 ANSWER 11 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1958:9094 CAPLUS

DN 52:9094

OREF 52:1628a-i,1629a-e

TI Disazo compounds

IN Feeman, James F.

PA Althouse Chemical Co.

DT Patent

LA Unavailable

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2795578		19570611	US	
GI	For diagram(s), see printed CA Issue.				
AB	<p>New disazo compds. of the general formula I, where Ar is a phenylene, biphenylene, a sulfonated stilbene radical, or a C₆H₄NHCOC₆H₄ or C₆H₄NHCNHC₆H₄ group, Ar' is a substituted phenyl group, x is 1, 2, or 3, and Y is CO, a fumaryl, a C(:O)C₆H₄C:O, or a 2-amino-1,3,5-triazinyl radical, are described. The amino group of the 2-aminotriazinyl radical may be substituted by a lower alkyl, a phenyl, or a lower alkylphenyl group. Ar may be substituted by 1-3 of the following groups: lower alkyl, lower alkoxy, halogen, CO₂H, OCH₂CO₂H, and SO₃H. Ar may be substituted by 1-3 of the following groups: lower alkyl, lower alkoxy, OCH₂CO₂H, acylamido, lower hydroxyalkyl, and lower hydroxyalkoxy. p-O₂NC₆H₄NH₂ is diazotized and coupled with 8,1,3,6-H₂NC₁₀H₄(SO₃H)₃. The resulting amino azo dye is oxidized, and the product treated with a reducing agent to give 2 - (p - aminophenyl) - 2H - naphtho[1.2]triazole - 4,7,9 - trisulfonic acid (II). II 50.0 is diazotized at 0° and coupled with m-MeC₆H₄NH₂ (III) 10.7 at pH 3.0-4.0. The resulting amino azo dye (IV) is treated at 25-40° and pH 6.5-7.5 in aqueous solution with COCl₂ to give a dye yellow-brown powder which dyed cotton and regenerated cellulose in yellow shades of excellent light- and wash-fastness. II 50.0 is diazotized and coupled at 0° with m-H₂NC₆H₄NHAc 15.0 parts at pH 3.0-4.5. The product is treated at 25-40° and pH 6.5-7.5 with COCl₂ to give a dye, brown-yellow powder. 5,2-Me(MeO)C₆H₃NH₂ 13.7 parts instead of III gives a similar dye, red-brown powder, orange on cotton. IV 61.8 in water 900 at 40° is treated with stirring with cyanuric chloride (V) 9.2 in Me₂CO 50 parts and simultaneously with aqueous Na₂CO₃ to maintain a pH of 6.5-7.5. The mixture is kept for 0.5 hr. at 40°, heated to 90°, treated with PhNH₂ 4.7 parts, heated 1 hr. at 90°, salted with NaCl, and filtered to yield a yellow-brown powder. Benzidine 18.4, 30% HCl 30, and water 200 are heated to 90°, cooled to 60°, treated with 30% HCl 20, cooled to 10°, and treated with NaNO₂ 14 in water during 5 min. The excess NaNO₂ is destroyed with H₂NSO₃H, the mixture cooled internally with ice to 0°, treated with NaHCO₃ 30, pyridine 25, and 8,1,3,6-H₂NC₁₀H₄(SO₃H)₃ 42 in water 100 while adding sufficient Na₂CO₃ to maintain a pH of 7.5. The solution is kept for 16 hrs. at 0-5°, adjusted to pH 5.5 with glacial AcOH 25, treated with III 12 in water 25 and 30% HCl 12, allowed to warm to 20° during 3 hrs., treated with NaOH 28, heated to 90°, treated with CuSO₄.5H₂O 50, heated for 1 hr. at 90°, treated with 30% HCl 100 parts, and filtered. The intermediate amino azo compound (VI) is dissolved</p>				

in water 1500 with NaOH to give a pH of 11, heated to 95°, treated with activated C 30 and siliceous filtering aid 50 parts, and filtered. The filtrate is salted with NaCl and filtered, and the residue treated in water at 25-50° and pH 6.5-7.5 with COCl₂ and salted with NaCl to give a dye, **yellow**-brown powder, **yellow** on cotton.

4-Amino-4'-nitro-2,2'-stilbenedisulfonic acid is diazotized and coupled with 6,1,3-H₂NC₁₀H₅(SO₃H)₂. The resulting nitroamino azo compound is oxidized with cupric ammonium hydroxide, and the product reduced with Fe powder in dilute AcOH to yield 2-[4-(4-amino-2-sulfoethyl)-3-sulphophenyl]-2H-naphtho[1,2]triazole-6,8-disulfonic acid (VII) which diazotized, coupled with III, and finally treated with COCl₂ gives a dye, orange-brown powder. VII is diazotized and coupled with anilinomethanesulfonic acid, and then treated with COCl₂ in the usual manner to give a dye, **yellow**-brown powder. If the 5-monosulfo analog of VII is diazotized instead of VII, then is coupled with III, and treated with COCl₂ a dye, **yellow**-brown powder, is formed. VII 68.3 is diazotized and coupled with III 10.7. The product is treated with stirring with fumaroyl chloride 7.65 in C₆H₆ 40 parts to give a dye, orange-brown powder. VII 68.3 is diazotized and coupled with III 10.7. The resulting amino azo compound is treated in neutral aqueous solution at 40° with V 9.2 in Me₂CO 50 parts and aqueous Na₂CO₃. The resulting product is treated in the usual manner with PhNH₂ 4.7 parts to give a dye, orange-brown powder. VII 68.3 is diazotized, couple with 5,2-Me(MeO)C₆H₃NH₂ 13.7 parts, and treated with COCl₂ to give a dye, red-brown powder. Diazotized VII 68.3, m-H₂NC₆H₄NHAc 15.0 parts, and COCl₂ give a dye, orange-brown powder. The 7,9-disulfonic isomer of VII 68.3, diazotized and treated with III 10.7 parts, and COCl₂ give a dye, **yellow**-brown powder. The 3,6-disulfonic isomer (VIII) of VII gives similarly a dye, orange-brown powder. VIII, III, and fumaroyl chloride give a dye, orange-brown powder. VIII, III, and V give a dye, orange-brown powder. II 50 in water 500 is treated at 75° with a nonionic surface-active dispersing agent 1, NaOAc 25, and p-O₂NC₆H₄COCl 25 with stirring, kept 10 min. at 75°, heated to 95°, treated during 15 min. with Fe powder 50 parts, heated 0.5 hr. at 95-100°, adjusted with Na₂CO₃ to pH 9.0, and filtered. The filtrate is treated with 10% NaCl, cooled, and filtered. The residue is dissolved in water 500 parts and reprecipitated at 25° and pH 1.5 with 30% HCl. The resulting intermediate in water 500 is diazotized at pH 7 with NaNO₂ 4 parts, the mixture treated with 30% HCl 20, cooled internally with ice to 0° for 2 hrs., the excess NaNO₂ destroyed with H₂NSO₃H, the mixture treated with III 6, adjusted with NaOAc to pH 3.0-3.5, and filtered at 20° and pH 1.5. The filter cake is dissolved in water 500, treated with activated C 10 parts at 70°, filtered, and treated with COCl₂ at pH 6.5-7.5 and 25-60°, and the product isolated by filtration to give a dye, orange powder. VI 69.5 is treated with fumaroyl chloride 7.65 in C₆H₆ 40 parts and dropwise with aqueous Na₂CO₃ at 25-30° to give a dye, **yellow**-brown powder. VI 69.5 in water is treated with V 9.2 in Me₂CO 50, and aqueous Na₂CO₃. The intermediate is treated with PhNH₂ 4.7 parts to give in the usual manner a dye, **yellow**-brown powder. All I yielded to orange shades of excellent light- and wash-fastness on cotton and regenerated cellulose fibers.

L9 ANSWER 12 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1958:9093 CAPLUS

DN 52:9093

OREF 52:1628a-i,1629a-e

TI Disazo compounds

IN Feeman, James F.

PA Althouse Chemical Co.

DT Patent

LA Unavailable

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2795577		19570611	US	
GI	For diagram(s), see printed CA Issue.				
AB	<p>New disazo compds. of the general formula I, where Ar is a phenylene, biphenylene, a sulfonated stilbene radical, or a C₆H₄NHCOC₆H₄ or C₆H₄NHCONHC₆H₄ group, Ar' is a substituted phenyl group, x is 1, 2, or 3, and Y is CO, a fumaryl, a C(:O)C₆H₄C(:O), or a 2-amino-1,3,5-triazinyl radical, are described. The amino group of the 2-aminotriazinyl radical may be substituted by a lower alkyl, a phenyl, or a lower alkylphenyl group. Ar may be substituted by 1-3 of the following groups: lower alkyl, lower alkoxy, halogen, CO₂H, OCH₂CO₂H, and SO₃H. Ar may be substituted by 1-3 of the following groups: lower alkyl, lower alkoxy, OCH₂CO₂H, acylamido, lower hydroxyalkyl, and lower hydroxyalkoxy. p-O₂NC₆H₄NH₂ is diazotized and coupled with 8,1,3,6-H₂NC₁₀H₄(SO₃H)₃. The resulting amino azo dye is oxidized, and the product treated with a reducing agent to give 2 - (p - aminophenyl) - 2H - naphtho[1.2]triazole - 4,7,9 - trisulfonic acid (II). II 50.0 is diazotized at 0° and coupled with m-MeC₆H₄NH₂ (III) 10.7 at pH 3.0-4.0. The resulting amino azo dye (IV) is treated at 25-40° and pH 6.5-7.5 in aqueous solution with COCl₂ to give a dye yellow-brown powder which dyed cotton and regenerated cellulose in yellow shades of excellent light- and wash-fastness. II 50.0 is diazotized and coupled at 0° with m-H₂NC₆H₄NHAc 15.0 parts at pH 3.0-4.5. The product is treated at 25-40° and pH 6.5-7.5 with COCl₂ to give a dye, brown-yellow powder. 5,2-Me(MeO)C₆H₃NH₂ 13.7 parts instead of III gives a similar dye, red-brown powder, orange on cotton. IV 61.8 in water 900 at 40° is treated with stirring with cyanuric chloride (V) 9.2 in Me₂CO 50 parts and simultaneously with aqueous Na₂CO₃ to maintain a pH of 6.5-7.5. The mixture is kept for 0.5 hr. at 40°, heated to 90°, treated with PhNH₂ 4.7 parts, heated 1 hr. at 90°, salted with NaCl, and filtered to yield a yellow-brown powder. Benzidine 18.4, 30% HCl 30, and water 200 are heated to 90°, cooled to 60°, treated with 30% HCl 20, cooled to 10°, and treated with NaNO₂ 14 in water during 5 min. The excess NaNO₂ is destroyed with H₂NSO₃H, the mixture cooled internally with ice to 0°, treated with NaHCO₃ 30, pyridine 25, and 8,1,3,6-H₂NC₁₀H₄(SO₃H)₃ 42 in water 100 while adding sufficient Na₂CO₃ to maintain a pH of 7.5. The solution is kept for 16 hrs. at 0-5°, adjusted to pH 5.5 with glacial AcOH 25, treated with III 12 in water 25 and 30% HCl 12, allowed to warm to 20° during 3 hrs., treated with NaOH 28, heated to 90°, treated with CuSO₄.5H₂O 50, heated for 1 hr. at 90°, treated with 30% HCl 100 parts, and filtered. The intermediate amino azo compound (VI) is dissolved</p>				

in water 1500 with NaOH to give a pH of 11, heated to 95°, treated with activated C 30 and siliceous filtering aid 50 parts, and filtered. The filtrate is salted with NaCl and filtered, and the residue treated in water at 25-50° and pH 6.5-7.5 with COCl₂ and salted with NaCl to give a dye, **yellow**-brown powder, **yellow** on cotton.

4-Amino-4'-nitro-2,2'-stilbenedisulfonic acid is diazotized and coupled with 6,1,3-H₂NC₁₀H₅(SO₃H)₂. The resulting nitroamino azo compound is oxidized with cupric ammonium hydroxide, and the product reduced with Fe powder in dilute AcOH to yield 2-[4-(4-amino-2-sulfoethyl)-3-sulfophenyl]-2H-naphtho[1,2]triazole-6,8-disulfonic acid (VII) which diazotized, coupled with III, and finally treated with COCl₂ gives a dye, orange-brown powder. VII is diazotized and coupled with anilinomethanesulfonic acid, and then treated with COCl₂ in the usual manner to give a dye, **yellow**-brown powder. If the 5-monosulfo analog of VII is diazotized instead of VII, then is coupled with III, and treated with COCl₂ a dye, **yellow**-brown powder, is formed. VII 68.3 is diazotized and coupled with III 10.7. The product is treated with stirring with fumaroyl chloride 7.65 in C₆H₆ 40 parts to give a dye, orange-brown powder. VII 68.3 is diazotized and coupled with III 10.7. The resulting amino azo compound is treated in neutral aqueous solution at 40° with V 9.2 in Me₂CO 50 parts and aqueous Na₂CO₃. The resulting product is treated in the usual manner with PhNH₂ 4.7 parts to give a dye, orange-brown powder. VII 68.3 is diazotized, couple with 5,2-Me(MeO)C₆H₃NH₂ 13.7 parts, and treated with COCl₂ to give a dye, red-brown powder. Diazotized VII 68.3, m-H₂NC₆H₄NHAc 15.0 parts, and COCl₂ give a dye, orange-brown powder. The 7,9-disulfonic isomer of VII 68.3, diazotized and treated with III 10.7 parts, and COCl₂ give a dye, **yellow**-brown powder. The 3,6-disulfonic isomer (VIII) of VII gives similarly a dye, orange-brown powder. VIII, III, and fumaroyl chloride give a dye, orange-brown powder. VIII, III, and V give a dye, orange-brown powder. II 50 in water 500 is treated at 75° with a nonionic surface-active dispersing agent 1, NaOAc 25, and p-O₂NC₆H₄COCl 25 with stirring, kept 10 min. at 75°, heated to 95°, treated during 15 min. with Fe powder 50 parts, heated 0.5 hr. at 95-100°, adjusted with Na₂CO₃ to pH 9.0, and filtered. The filtrate is treated with 10% NaCl, cooled, and filtered. The residue is dissolved in water 500 parts and reprecipitated at 25° and pH 1.5 with 30% HCl. The resulting intermediate in water 500 is diazotized at pH 7 with NaNO₂ 4 parts, the mixture treated with 30% HCl 20, cooled internally with ice to 0° for 2 hrs., the excess NaNO₂ destroyed with H₂NSO₃H, the mixture treated with III 6, adjusted with NaOAc to pH 3.0-3.5, and filtered at 20° and pH 1.5. The filter cake is dissolved in water 500, treated with activated C 10 parts at 70°, filtered, and treated with COCl₂ at pH 6.5-7.5 and 25-60°, and the product isolated by filtration to give a dye, orange powder. VI 69.5 is treated with fumaroyl chloride 7.65 in C₆H₆ 40 parts and dropwise with aqueous Na₂CO₃ at 25-30° to give a dye, **yellow**-brown powder. VI 69.5 in water is treated with V 9.2 in Me₂CO 50, and aqueous Na₂CO₃. The intermediate is treated with PhNH₂ 4.7 parts to give in the usual manner a dye, **yellow**-brown powder. All I yielded to orange shades of excellent light- and wash-fastness on cotton and regenerated cellulose fibers.

L9 ANSWER 13 OF 13 CAPLUS COPYRIGHT 2004 ACS on STN

AN 1950:35803 CAPLUS

DN 44:35803

OREF 44:6842c-h

TI 3-Phenylindones. I. The synthesis of 6-chloro-3-(p-chlorophenyl)-1-indenone and some related compounds

AU Feeman, James F.; Amstutz, E. D.

CS Lehigh Univ., Bethlehem, PA

SO Journal of the American Chemical Society (1950), 72, 1522-6

CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA Unavailable

OS CASREACT 44:35803

AB Since it was believed that substitution of the conjugated system -C:C:C:O for the Cl3CMe grouping in the DDT mol., either as a fused ring system or in the form of a substituted acrylic ester, might be expected to produce compds. of value as selective insecticides, the 3-phenylindone structurally related to DDT has been prepared (p-ClC6H4)2CO (100.44 g.) and 133.6 g. BrCH2CO2Et in 300 mL. each of C6H6 and PhMe, treated with 65.4 g. 30-mesh Zn (cf. Fieser and Johnson, C.A. 34, 2838.9), refluxed 5 h., poured onto 500 g. ice and 200 mL. 20% H2SO4, and the product refluxed 2 h. with 44.8 g. KOH in 100 mL. H2O and 1 l. 95% EtOH, give 92.3% β,β -bis(p-chlorophenyl)hydracrylic acid (I), m. 197.5-8.5° (decomposition, m.ps. corrected); Et ester, m. 101-1.6°; Me ester, m. 125-6° (35.2% from BrCH2CO2Me). I (86 g.) in 700 mL. absolute EtOH, treated 4 h. with anhydrous HCl (boiled the last hr.), gives 83.5% of the Et ester (II), m. 64-5°, of (p-ClC6H4)2C:CHCO2H (III), m. 176-8° (prepared in 79.4% yield from 73.8 g. I and 500 mL. concentrated H2SO4 on standing 30 min. at room temperature). Hydrogenation of II over 5% Pd-C in EtOH at room temperature/2 atmospheric gives 85.5% of the Et ester, b7 205-9°, of (p-ClC6H4)2CHCH2CO2H (IV), m. 189-91°. III (8.79 g.) warmed with 7.14 g. SOCl2 (1 drop C5H5N), and the acid chloride in 25 mL. CS2 treated with 8 g. AlCl3 and warmed 20 h. on the steam bath, gives 60.6% 6-chloro-3-(p-chlorophenyl)indone (V), **yellow**, m. 160-2° (decomposition); 2,4-dinitrophenylhydrazone, dark red, m. 290-1° (decomposition). I or its Et ester with 10 mL. oleum (15% SO3) at room temperature gives 42.5% V. In like manner, IV gives 77.7% (crude) 6-chloro-3-(p-chlorophenyl)-1-indanone (VI), salmon, m. 117-18.5°; 2,4-dinitrophenylhydrazone, orange-red, m. 259-60°. Oxidation of V or VI with CrO3 in AcOH gives 5-chloro-2-(p-chlorobenzoyl)benzoic acid, m. 158-9°. Clemmensen reduction of VI yields 81.8% 5-chloro-1-(p-chlorophenyl)indan, b3 170-2°. V (1 g.) in 10 mL. CHCl3, treated with 0.58 g. Br, gives 78.4% of the 2-Br derivative, orange, m. 209-11°; 2,4-dinitrophenylhydrazone, dark orange, m. 257-8°. V and Cl in CHCl3 give the 2,6-di-Cl derivative (VII), orange, m. 196.5-7.6°; 2,4-dinitrophenylhydrazone, red, m. 270-3°. Further action of Cl on VII gives 2,2,3,6-tetrachloro-3-(p-chlorophenyl)-1-indanone, **yellow**, m. 150.6-2°.

=>